Fabrication of x-ray gratings by direct write mask-less lithography

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ABSTRACT

Fabrication of diffraction grating for x-rays is a very challenging problem due to the exacting requirements of surface quality, groove position, and groove profile. Traditional fabrication techniques have significant limitations and do not cover all the necessary requirements. For example, classical holographic recording is limited in the type of groove patterns that can be produced. This is particularly important in the design of wide aperture high resolution spectrometers, where aberration correction using complex groove patterns is necessary. We are pioneering the use of direct-write mask-less optical lithography to make grating patterns of arbitrary complexity. In this work we report on the first results from our direct-write mask-less approach, including quality assessment of the patterns using interferometric techniques.

Key words: diffraction grating, x-rays, mask-less lithography, optical metrology, AFM, plasma etch.

Introduction

There is a great demand for high precision x-ray diffraction gratings for synchrotron soft x-ray beamlines and spectrometers [1]. Due to the very high source brightness of today's synchrotron and Free Electron Laser (FEL) sources, high quality gratings are essential to meet the exacting requirements in terms of resolution and efficiency. To meet these requirements the gratings should have a large clear aperture, precise groove position, and optimized groove shape and profile.

Variable Line Spacing (VLS) gratings are often used in brightness preserving optical schemes since they combine both diffraction and imaging abilities in one optical element. The groove placement accuracy is crucial for these gratings since a linear variation of groove density along the grating length defines focusing and a quadratic term provides aberration control.

Diamond ruling and holographic recording are the two main techniques for soft x-ray grating fabrication. The diamond ruling process, as used for fabrication of blazed gratings including VSL, is a very slow and expensive process. Fabrication of one typical soft x-ray grating can take a month or even more [2]. Such a long process imposes extremely tight requirements on environmental stability which are very difficult to fulfill. VLS gratings can also be recorded holographically with some limitations though coming from a limited flexibility of the technique. Often a grating manufacturer can not provide a groove density variation with the precision required and final grating specifications are a compromise between the original grating design and vendor capabilities. This is obviously a limiting factor on a performance of a soft x-ray beamline. All these issues stimulate our interest in novel non-traditional grating fabrication techniques which address these fundamental limitations of traditional grating fabrication methods. In this work we investigate applicability of Direct Write Lithography (DWL) technique for making diffraction gratings for soft x-rays.

Modern design methods are allowing us to explore complicated groove patterns that allow aberration correction, even for extremely large angular apertures. This is particularly important for photon-hungry applications such as x-ray fluorescence, where we need to collect large apertures, but preserve high resolution. Reflective zone plates (RZP) consist of elliptical grooves [3] and are useful for many applications where the highest throughput and simplest optical systems are needed. One such example is femtosecond ultrafast x-ray slicing experiments, where total source flux is very low, and the highest possible efficiency is needed. This is again an application where DWL written structures can be very useful.

Our goal is to investigate and exploit new high precision patterning techniques for grating patterns of arbitrary complexity. In this work we have used Direct Write Lithography (DWL) as a grating fabrication technique. The pattern is recorded by scanning of a focused laser beam over a grating substrate coated with a resist. A precise interferometrically controlled stage provides high accuracy in position of the patterned features. The method is completely flexible in terms of pattern complexity and seems promising for making VLS and RPZ structures. Unlike e-beam lithography, the DWL method can be very fast, minimizing the opportunity for low frequency noise. After resist development the pattern can be transferred to a substrate by ion sputtering or plasma etch resulting in a lamellar grating. Alternatively, the pattern can be transferred to a hard mask layer to apply anisotropic wet etching process to make a saw-tooth grating afterwards [6,7]. The focus of this work is to assess the patterning positioning of DWL, in the light of the very high precision needed for diffraction gratings.

Patterning of the gratings with DWL66 and DWL2000 tools

We used two DWL tools from Heidelberg [5] to generate test grating patterns for grating quality assessment. Most of test exposures were performed using a Heidelberg DWL66 machine available in the Bimolecular Nanotechnology Center (BNC) of the University of California in Berkeley (UCB) [8]. This however was an older generation instrument which was not expected to be precise enough in terms of the very high requirements for groove accuracy, but afforded us an easy way to test out the technology. Final exposures were performed using a DWL2000 system installed at the company site in Heidelberg, Germany. This advanced tool has a much more precise stage, a smaller minimum feature size, sophisticated scanning algorithms which provide a much higher writing speed, better focusing control, environment control, and was expected to provide a much higher quality of the grating patterns. Detailed specification of DWL66 and DWL2000 machines tools can be found on the company website [4].

Silicon substrates with a diameter of 100 mm and thickness of 5 mm were used as grating substrates. For preliminary experiments regular 4” silicon wafers and 4 mm thick glass mask blanks were used as well. The substrates were spin-coated with 550 nm thick S1805 resist and prebaked at 115ºC as recommended.

Grating patterns with a groove density of 300 lines/mm and 600 lines/mm were recorded using DWL66 and DWL2000 tools. The constant groove density patterns are convenient for groove position accuracy characterization via wavefront measurements. We also fabricated VLS gratings which will be tested and reported soon. The typical size of the patterns was 60 mm by 30 mm (Fig.1). The grooves of the patterns were aligned along the short side of the patterns. We used different patterning schemas for the different machines. DWL66 tool exploited a simple writing algorithm with fast scanning along the groove direction and slow motion of the substrate in the direction perpendicular to the grooves. The drawback of the DWL66 approach is fairly low writing speed which results in typical exposure time of dozens hours. This not only reduces the tool throughput but makes the machine vulnerable to low frequency variations of environmental condition.

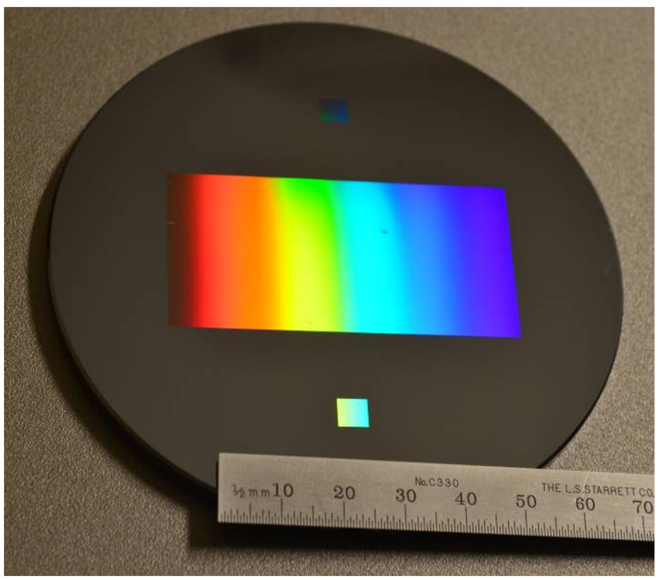

Fig. 1. A 600 lines/mm diffraction grating fabricated by DWL.

A much faster scan approach is implemented for DWL2000 machine. Scanning along the grooves is performed by an acoustic optical deflection system and was combined with stage motion in the perpendicular direction of the grating length. One pass of the stage resulted in a 160 μm wide stripe, so about 190 stripes were written to cover the 30 mm wide pattern. Since the optical deflection system is very fast, the duration of the exposure was shortened down to 30 min. This is tremendous improvement as compared to the old DWL66 approach and traditional lithography. An obvious concern regarding the stripe approach is possible stitching errors of the grooves at the stripe boundaries. This problem is being addressed in our present work.

Post exposure processing and groove shaping

After exposure the patterns were inspected with SEM in order to verify readiness of the samples for the plasma etch step. Both top view and cross section of the resist stripes were investigated. In the latter case test we used patterns on a regular 100 mm diameter wafers which were cleaved to make cross-section samples. Since an anti-reflective coating (ARC) was not used in these experiments the resist profile exhibited wavy sidewalls caused by interference effects (Fig. 2a). The non-optimal resist profile causes significant line edge roughness. Also residual resist traces were observed in between the stripes. To remove the unwanted resist contamination, to improve the profile of the resist stripes, and tune the duty-cycle ratio of the patterns to the optimal value of 0.4 -0.5, we used low power Oxygen plasma etch. After the Oxygen plasma treatment the resist stripes had a much better profile and significantly reduced line edge roughness (LER) as seen in Fig. 2b. Then the grating grooves were etched by a CF_4 reactive plasma etch of

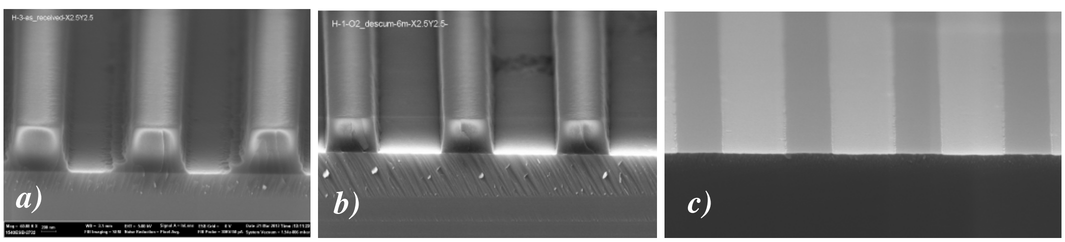

Fig. 2. Resist pattern after exposure and development (a), after Oxygen plasma etch (b), and 600 lines/mm silicon grating after resist removal and cleaning (c).

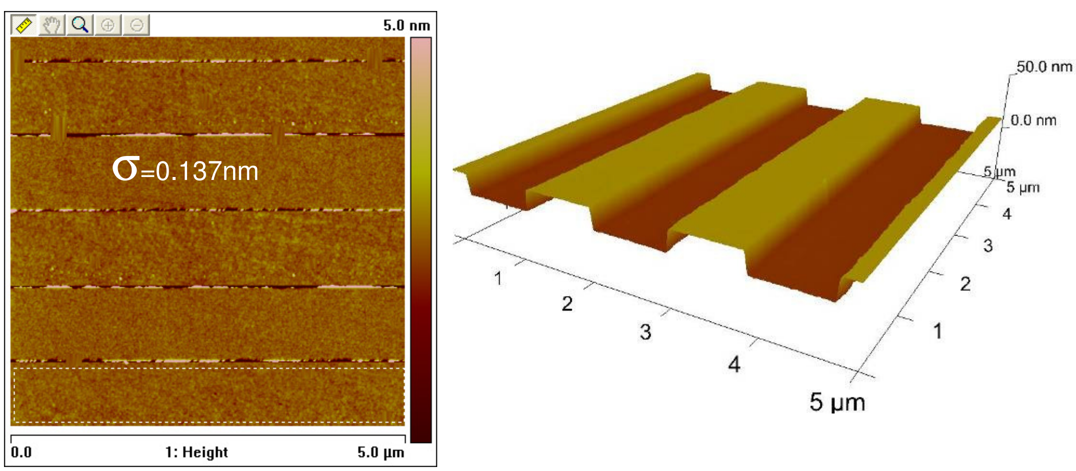

Fig. 3. Top (a) and 3D (b) AFM images of a 600 lines/mm Silicon diffraction grating: reactive plasma etch provides precise groove depth control and a smooth surface of the grating grooves.

silicon. Since grazing incidence x-ray gratings typically have very shallow grooves of 5-30 nm in depth, a very precise control of the etch rate is required. CF_4 plasma etching which is usually used for silicon nitride and silicon oxide etch, etches Si very slowly and hence suits our purposes perfectly. Finally, the resist was removed and the gratings were cleaned with Piranha solution ($H_2SO_4 + H_2O_2$). SEM image of a test grating sample after plasma etch and cleaning is shown in Fig. 2c. AFM inspection of the gratings confirmed the groove depth and quality of the surface of the grooves after the plasma etch process (Fig. 3).

Assessment of grating quality by wavefront measurements

One of the main challenges of x-ray grating fabrication technique is the need to have high placement accuracy of the groove positions. A common criterion for affordable groove displacement for optical diffraction gratings is that the wavefront distortions caused by groove position errors should not exceed $\lambda/10$, where λ is a wavelength of illumination. This leads to maximum allowed errors of the absolute displacement of a groove from its ideal position of $\varepsilon < d/10$, where d is a grating period [9]. Note that the criterion does not depend on the wavelength, allowing assessment of x-ray gratings with visible light interferometry.

The wavefront measurements of the grating patterns were performed using a Zygo GPI 6” Fizeau interferometer. In this type of measurements a grating is set in Littrow geometry and wavefront of a diffracted wave was tested against a reference wavefront of the interferometer (Fig. 4). A fringe pattern formed as a result of interference of the reference and diffracted waves is converted into a wavefront error map. An ideal constant groove density grating should have flat wavefront of the diffracted wave. When a groove is displaced from its correct position by an amount ε, the corresponding error of the wavefront, σ, is:

$$\sigma = \pm 2\varepsilon sin\beta \qquad (1)$$

where the sign of the error depends on the right/left direction of the displacement with respect to an ideal position or on a direction of illumination which corresponds to diffraction into a positive or negative diffraction order.

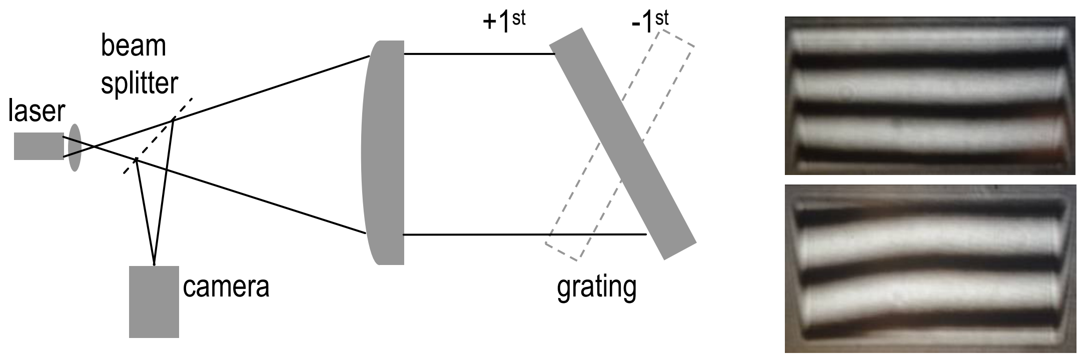

Fig. 4. A schematic of wavefront measurements with a Fizeau interferometer (at left); examples of fringe patterns for first positive and negative diffraction orders are shown at right up and bottom respectively.

In addition to the groove position errors, a waterfront is perturbed by substrate surface relieve as well as interferometer optics imperfections. To exclude contributions of these additional factors two error maps corresponding to positive and negative order diffraction were recorded and then subtracted from each other. Displacement of grating grooves results in wavefront errors of opposite signs for the negative and positive orders, while the errors caused by surface relieve as well as by instrument imperfections have the same sign and cancel after the subtraction. In this way the differential measurement yields doubled wavefront distortions caused solely by the groove position errors. Since the interferometer software is optimized for substrate surface relief measurements the wavefront data are divided by 2 automatically, and the diffracted wavefront topography data obtained as was described above can be directly used for groove displacement calculations using formula (1).

The differential technique is very useful for routine assessment of test patterns since it does not require very high quality of the grating substrates. In fact most of preliminary measurements were performed for regular quality Si wafers or glass mask blanks which had a fairly wavy surface. Despite the surface of the test substrates producing significant perturbations of the wavefront, the latter were successfully removed by the subtraction procedure allowing elimination of the contribution from the substrate imperfections and the tool systematic errors.

Another possible way to take into account the surface relieve is to perform zero order measurements and subtract the relief from the diffraction wavefront. We found this works well also, but this method requires a correction of the wavefront image size by the angle of rotation of the grating. For low groove density gratings the angle correction was marginal (less than a pixel size of the interferometer camera) but for high groove density gratings an appropriate correction must be done for zero/diffraction order measurements. At the same time there is no need to do the correction for +/- order diffraction since the image size in this case is the same. Moreover, as it was mentioned above the positive/negative order wavefront difference is twice as big as the positive-zero order difference and hence provides better sensitivity of the measurements. Additional sensitivity enhancement can be obtained for high order diffraction, however it requires rotation at a larger angle and may affect image resolution along the grating length direction (perpendicular to the grooves). Moreover, since efficiency of diffraction typically reduces with the number of an order, use of high order diffraction can result in undesirable noise in the wavefront image.

A grating #1 with groove density of 300 lines/mm was patterned with DWL66 machine. Writing lens with working distance of 4 mm was used. Stage motion control was performed with a 20 nm grid. A differential error map for the +/- diffraction orders, shown in Fig. 1, reveals a substantial waviness of the diffracted wavefront. The direction of the waviness coincides with the low rate scan direction (along the grating length and perpendicular to the grooves). The observed “one-dimensional” waterfront topography is caused by errors in groove position rather than groove curvature. Since the DWL66 machine has a basic enclosure which does not provide a proper climate control the errors are probably related to environmental instabilities during the long-term writing process. The groove position errors reach up to 500 nm or d/6 as seen in Fig. 1c where groove displacements calculated for a central cross-section of the wavefront map is shown. Such a pattern does not meet quality requirements for x-ray gratings, VLS in particular. A red curve in Fig. 1d depicts a second order variation of a VLS grating which is used in a monochromator of Maestro beamline of ALS. The groove position errors of the pattern #1 result in significant variation of local groove density which exceeds the quadratic component and hence such a grating is not capable to provide a correction of coma aberrations, which is vital for the beamline. According the grating quality criterion the groove accuracy should be improved by a factor of 2. However, Fig. 5d shows that even 2-fold reduction of groove density noise would barely help with control of the quadratic term. This demonstrates that requirements for x-ray gratings can be much more stringent than criteria accepted for visible light optics.

Advanced capabilities of the DWL2000 machine provide much more stable operation allowing higher groove density and higher quality of gratings. The wavefront errors for a 600 lines/mm pattern shown in Fig. 6a,b do not exceed 30 nm (p/v) which is less than half the λ/10 criteria. The groove displacement was estimated to be as low as 80 nm p/v or d/21. This indicates that gratings with groove density of up to 1200 lines/mm that have been patterned with the DWL2000 would successfully meet the optical criteria.

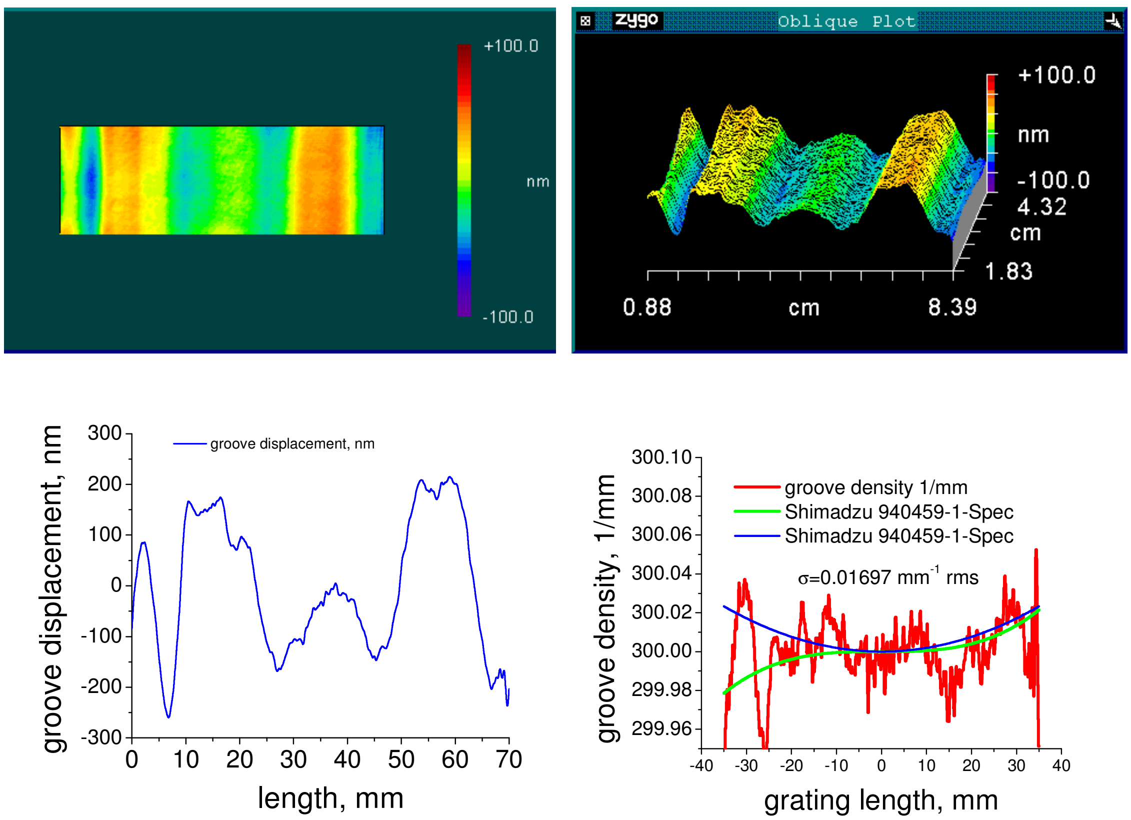

Fig. 5. Top (a) and 3D (b) view of the wavefront for the +/- 1st diffraction orders for a 300 lines/mm grating #1 fabricated with a DWL66 tool; groove displacement errors (c) and groove density variations (d) calculated for a central cross-section of the wavefront image. Blue and green curves in Fig. 5d depict second and third polynomial variation of groove density specified for a VLS diffraction grating installed in a monohromator of the Maestro beamline of the Advanced Light Source.

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Unlike DWL66 patterns the residual wavefront error map shown in Fig. 6 exhibits two-dimension variation of the surface height, which correlates to the writing algorithm of the DWL2000 tool. Note, that DWL2000 suggests many different algorithms of writing which will be tested in future including groove-by-groove writing over whole width of a pattern similar to DWL66 patterning. As it was mentioned above such algorithm excludes possible stitching errors.

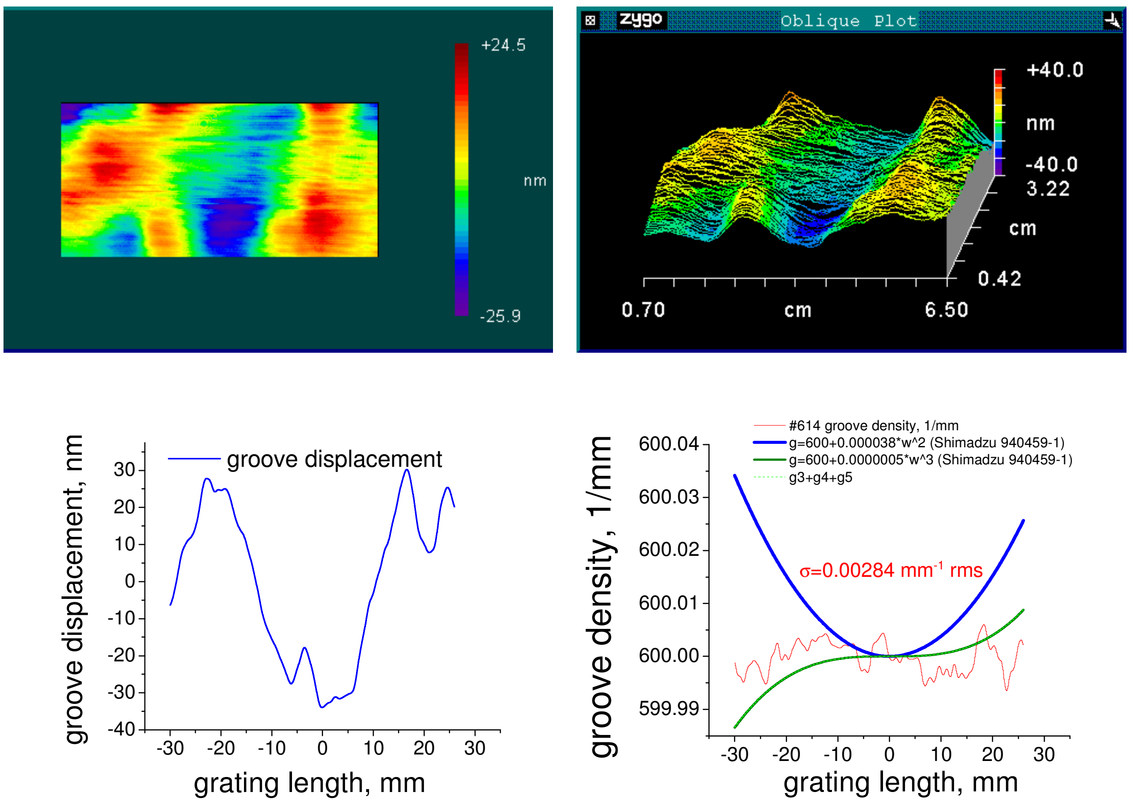

Fig. 6. Top (a) and 3D (b) view of the wavefront for the +/- 1st diffraction orders for a 600 lines/mm grating #2 fabricated with a DWL2000 tool; groove displacement errors (c) and groove density variations (d) calculated for a central cross-section of the wavefront image. Blue and green curves in Fig. 6d depict second and third polynomial variation of groove density specified for a VLS diffraction grating installed in a monochromator of the Maestro beamline at the Advanced Light Source.

Again, the groove density variation caused by groove displacement can be compared to requirements for a real VLS x-ray grating. The second-order polynomial variation of another Maestro grating with an average grove density of 600 lines/mm is shown in Fig. 6d by the blue curve. The green curve in Fig. 6d depicts a maximum allowed third-order polynomial variation which ideally should be zero. One can see that the errors in groove density of grating #2 do not exceed the third order tolerances for most of the grating area, except some central area. The random groove density noise is much smaller than the desired second-order variation of groove density. This confirms that the DWL approach is promising for practical x-ray grating fabrication.

Another comparison of quality of the DWL patterns can be made against gratings fabricated with an alternative technique, for example, by holography. Since whole the area of such gratings is recorded simultaneously these gratings should be free of time dependent factors like temporal stability, environmental issues etc. Results of wavefront measurements for a holographic Richardson grating with groove density of 1200 lines/mm are shown in Fig. 7. A surface of a wavefront has a characteristic saddle shape caused by non-ideal flatness of the waves used for holographic recording. However, these distortions are fairly small and caused by groove position errors as low as 40 nm p/v which is twice smaller than for the grating #2. This shows a room for improvement of the DWL technology for grating fabrication. Such an improvement combined with great flexibility of the DWL patterning in terms on groove density variation and a groove shape can make DWL a leading technology for x-ray grating fabrication.

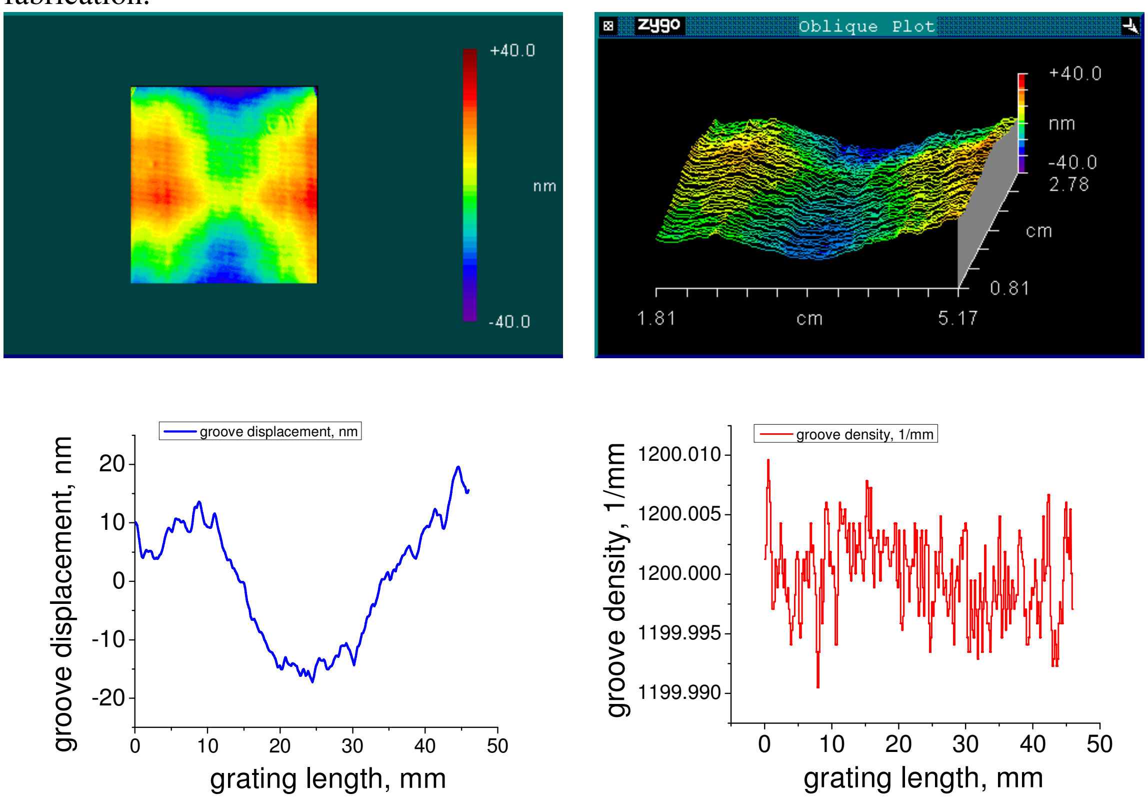

Fig. 7. Top (a) and 3D (b) view of the wavefront for the +/- 1st diffraction orders for a 1200 lines/mm commercial holographic grating (Richardson); groove displacement errors (c) and groove density variations (d) calculated for a central cross-section of the wavefront image.

Summary

We demonstrated successful fabrication of x-ray grazing incidence diffraction gratings using a DWL technique. Several grating prototypes were fabricated and extensively characterized by interferometric methods. Wavefront measurements of the gratings showed that modern DWL tools are capable of providing a very low level of errors in the position of grating grooves. The precision of the DWL gratings approaches that of holographic gratings and may satisfy quality requirements for many x-ray spectroscopy applications. Further development and improvement of the DWL method combined with the great flexibility in choice of pattern parameters such as groove density variation, groove shape, and a high writing speed of the commercial tools should make DWL a leading technology for x-ray grating fabrication. This would make a significant impact on beamline design, in terms of allowing designs with higher throughput and resolution.

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